#### APPENDIX F

The following method QC criteria, equations, and definitions apply to data generated according to the USEPA CLP Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration, OLM04.3, Exhibit D Pesticides/Aroclors.

#### SECTION I: PRESERVATION & HOLDING TIME CRITERIA

Refer to <u>Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses</u>, Part III, Section PEST/PCB-I-B for preservation and holding time data validation criteria.

#### SECTION II: GC/ECD INSTRUMENT PERFORMANCE CHECK CRITERIA

Refer to <u>Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses</u>, Part III, Section PEST/PCB-II-B for GC/ECD Instrument Performance Check data validation criteria and the following method GC/ECD instrument performance check QC criteria:

## Resolution Check Mixture

A Resolution Check Mixture containing the following analytes must be analyzed at the beginning of the initial calibration sequence. The resolution between two adjacent peaks in the Resolution Check mixture must be greater than or equal to 60.0%.

Analyte	Concentration (ng/mL)	
gamma-Chlordane	10.0	
Endosulfan I	10.0	
4,4'-DDE	20.0	
Dieldrin	20.0	
Endosulfan sulfate	20.0	
Endrin ketone	20.0	
Methoxychlor	100.0	
Tetrachloro-m-xylene (surrogate)	20.0	
Decachlorobiphenyl (surrogate)	20.0	

**Resolution Check** - The % Resolution is calculated using the following equation:

% Resolution = 
$$\frac{V}{H}$$
 x 100

Where,

V = Depth of valley between two peaks. The depth of the valley is measured along a vertical line from the level of the apex of the shorter peak to the floor of the valley between the two peaks.

H = Height of the shorter peak.

#### Example

IF:

Height of Peak A = 2560 Height of Peak B = 1435

The resolution (depth of the valley) between the two peaks must be at least 60.0% of the shorter peak or Peak B in this situation (0.6 x 1435 = 861). If the resolution between Peak A and Peak B is less than 861, the data will need to be qualified as indicated in Tables Pest/PCB-II-1 and Pest/PCB-II-2.

## Performance Evaluation Mixture

A Performance Evaluation Mixture (PEM) containing the analytes listed below must be analyzed at the beginning of the initial calibration sequence, immediately after the Resolution Check mixture and at the end of the initial calibration sequence. It must also be analyzed once during every 24 hours of the analytical sequence as part of the calibration verification. The resolution between two adjacent peaks in the PEM must be greater than or equal to 90.0%. The percent breakdown for both DDT and Endrin in each PEM must be less than or equal to 20.0% for both GC columns. The combined percent breakdown for DDT and Endrin in each PEM must be less than or equal to 30.0% for both GC columns.

Analyte	Concentration (ng/mL)	
gamma-BHC	10.0	
alpha-BHC	10.0	
4,4'-DDT	100.0	
beta-BHC	10.0	
Endrin	50.0	
Methoxychlor	250.0	
Tetrachloro-m-xylene (surrogate)	20.0	
Decachlorobiphenyl (surrogate)	20.0	

**Percent Difference (% D)** - The % D of the calculated amount (amount found) and the nominal amount (amount added) for each of the single component pesticides and surrogates in the PEM analysis of the initial calibration sequence on each GC column must be less than or equal to  $\pm 25.0\%$ . The % D is calculated using the following equation:

% Difference = 
$$\frac{C_{calc} - C_{nom}}{C_{nom}} \times 100$$

Where,

C<sub>calc</sub> = Calculated concentration of each analyte from the analyses of the calibration standards.

 $C_{nom}$  = Nominal concentration of each analyte.

## 4,4'-DDT/ENDRIN BREAKDOWN CALCULATIONS

% Breakdown DDT = 
$$\frac{Amount found (ng) (DDD + DDE)}{Amount (ng) of DDT injected} \times 100$$

% Breakdown Endrin = 
$$\frac{Amount found(ng) (Endrin Aldehyde + Endrin Ketone)}{Amount (ng) of Endrin injected} \times 100$$

Combined % Breakdown = % Breakdown DDT + % Breakdown Endrin

#### SECTION III: INITIAL CALIBRATION CRITERIA

Refer to <u>Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses</u>, Part III, Section PEST/PCB-III-B for initial calibration data validation criteria and the following method initial calibration QC criteria:

Each GC/ECD system must be initially calibrated using the following sequence:

## INITIAL CALIBRATION SEQUENCE

- 1. Resolution Check
- 2. Performance Evaluation Mixture
- 3. Aroclor 1016/1260
- 4. Aroclor 1221
- 5. Aroclor 1232
- 6. Aroclor 1242
- 7. Aroclor 1248
- 8. Aroclor 1254
- 9. Toxaphene
- 10. Low Point Standard A
- 11. Low Point Standard B
- 12. Midpoint Standard A
- 13. Midpoint Standard B
- 14. High Point Standard A
- 15. High Point Standard B
- 16. Instrument Blank
- 17. Performance Evaluation Mixture

## <u>Table App.F.III-1 - ANALYTES CONTAINED IN INDIVIDUAL STANDARD MIXTURE A AND INDIVIDUAL STANDARD MIXTURE B</u>

IND STD MIXTURE A	LOW POINT CONCENTRATION (ng/mL)
alpha-BHC	5.0
Heptachlor	5.0
gamma-BHC	5.0
Endosulfan I	5.0
Dieldrin	10.0

Endrin	10.0
4,4'-DDD	10.0
4,4'-DDT	10.0
Methoxychlor	50.0
Tetrachloro-m-xylene (surrogate)	5.0
Decachlorobiphenyl (surrogate)	10.0
IND STD MIXTURE B	LOW POINT CONCENTRATION (ng/mL)
beta-BHC	5.0
delta-BHC	5.0
Aldrin	5.0
Heptachlor epoxide	5.0
alpha-Chlordane	5.0
gamma-Chlordane	5.0
4,4'-DDE	10.0
Endosulfan sulfate	10.0
Endrin aldehyde	10.0
Endrin ketone	10.0
Endosulfan II	10.0

Tetrachloro-m-xylene (surrogate)

Decachlorobiphenyl (surrogate)

## Table App.F.III-2 - MULTICOMPONENT ANALYTE LOW POINT CONCENTRATIONS

5.0

10.0

MULTICOMPONENT ANALYTES	LOW POINT CONCENTRATIONS (ng/mL)
Aroclor 1016	100
Aroclor 1221	200
Aroclor 1232	100
Aroclor 1242	100
Aroclor 1248	100
Aroclor 1254	100
Aroclor 1260	100
Toxaphene	500

Multicomponent standards including the Aroclors and Toxaphene must be prepared individually except for Aroclor 1260 and Aroclor 1016 which may be combined in one standard mixture.

## RETENTION TIME WINDOW CALCULATION

In the initial calibration (low, mid, and high point) the absolute retention times (RTs) are measured for all single component pesticides, the surrogates, and at least three major peaks of each multicomponent analyte. The mean RTs for single component standards and surrogates are calculated as the average of the three values and the RTs for multicomponent analytes are based on one value.

A retention time window for each single component pesticide and surrogate and for three to five major peaks of each multicomponent analyte are calculated using the values in Table App.F.III-3.

The following equation is used when calculating a mean absolute retention time for each single component pesticide and surrogate:

$$\overline{RT} = \frac{\sum_{i=1}^{n} RT_{i}}{n}$$

Where,

RT = Mean absolute retention time of analyte.

RT<sub>i</sub> = Absolute retention time of analyte.

n = Number of measurements (3).

## Example

The retention time window is calculated by first taking the mean of the retention times from the low, mid, and high concentration of the individual standards in the initial calibration. For example the retention times for Endrin are:

Low - 9.86 Mid - 9.85

High - 9.86

Mean = 9.86

Since we know from Table App.F.III-3 that the retention time window for Endrin is  $\pm 0.07$ , we add and subtract 0.07 to and from the Mean to calculate the retention time window for Endrin from the initial calibration.

RT Window for Endrin = 9.79 - 9.93

Table App.F.III-3 - RETENTION TIME WINDOWS FOR ANALYTES AND SURROGATES

Analyte	Retention Time Window in Minutes
alpha-BHC	±0.05
beta-BHC	±0.05
gamma-BHC	±0.05
delta-BHC	±0.05
Heptachlor	±0.05
Aldrin	±0.05
alpha-Chlordane	±0.07
gamma-Chlordane	±0.07
Heptachlor Epoxide	±0.07
Dieldrin	±0.07
Endrin	±0.07
Endrin Aldehyde	±0.07
Endrin Ketone	±0.07

Analyte	Retention Time Window in Minutes
DDD	±0.07
DDE	±0.07
DDT	±0.07
Endosulfan I	±0.07
Endosulfan II	±0.07
Endosulfan Sulfate	±0.07
Methoxychlor	±0.07
Aroclors	±0.07
Toxaphene	±0.07
Tetrachloro-m-xylene (surrogate)	±0.05
Decachlorobiphenyl (surrogate)	±0.10

## CALIBRATION FACTOR CALCULATION

In the initial calibration, peak areas or peak heights are measured to determine the analyte Calibration Factor (CF). The Calibration Factor for each single component pesticide and surrogate and for each peak in the selected set of 3 to 5 major peaks for each multicomponent analyte is calculated using equation III-1. The mean CF for each single component pesticide and surrogate is calculated using equation III-2. Note: The single component pesticide mean CF is only used in calculating the % RSD and not for quantifying sample results.

Equation III-1 is as follows:

$$CF = \frac{Peak Area (height) of the standard}{Mass injected (ng)}$$

Equation III-2 is as follows:

$$\overline{CF} = \frac{\sum_{i=1}^{n} CF_{i}}{n}$$

Where,

CF = Mean calibration factor.

 $CF_i$  = Calibration factor.

n = Total number of values (3).

#### % RSD CALCULATION

Initial calibration data is used to assess the linearity of each GC/ECD system used for sample analysis. The linearity of the GC is assessed by calculating a % RSD of the calibration factors for each single component pesticide and surrogate.

Equation III-3 is as follows:

% 
$$RSD = \frac{SD_{CF}}{\overline{CF}} \times 100$$

Where,

$$SD_{CF} = \sqrt{\sum_{i=1}^{n} \frac{(CF_i - \overline{CF})^2}{(n-1)}}$$

CF = Mean calibration factor.

CF = Each individual Calibration Factor used to calculate the mean.

n = Total number of values (3).

% RSD = Percent Relative Standard Deviation.

 $SD_{CF}$  = Standard Deviation of the calibration factors.

## SECTION IV: CALIBRATION VERIFICATION CRITERIA

Refer to the <u>Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses</u>, Part III, Section PEST/PCB-IV-B for calibration verification data validation criteria and the following method calibration verification QC criteria:

The analyses of the instrument blanks, Performance Evaluation Mixtures (PEM), and the midpoint concentration of Individual Standard Mixtures A and B constitute the calibration verification. Sample data are not acceptable unless bracketed by acceptable analyses of instrument blanks, PEM, and both Individual Standard Mixtures A and B.

A valid analysis sequence is given below:

## 1. Sequence

Time	Injection #	Material Injected
	1 - 15	First 15 steps of the Initial Calibration
0 hr.	16	Instrument Blank* at the end of the Initial Calibration
	17	PEM at the end of the Initial Calibration
	18	First Sample
	o	
	O	Subsequent Samples
	O	
12 hr.	O	Last Sample
	1st injection past 12:00 hr.	Instrument Blank*
	2nd and 3rd injections	Individual Standard
	past 12:00 hr.	Mixtures A and B
	0	Sample
	0	•
	0	Subsequent Samples
	0	•
Another 12 hr.	O	Last Sample
	1st injection past 12:00 hr.	Instrument Blank*
	2nd injection past 12:00 hr.	Performance Evaluation Mixture
	0	Sample
	0	r .
	0	Subsequent Samples
	0	1
Another 12 hr.	0	Last Sample
	1st injection past	Instrument Blank*
	12:00 hr.	
	2nd and 3rd injections	Individual Standard
	past 12:00 hr.	Mixtures A and B
	0	Sample
	o	-
	o	Subsequent Samples
	etc.	

<sup>\*</sup>The instrument blank contains only the surrogate analytes: Tetrachloro-m-xylene and Decachlorobiphenyl.

**PERCENT DIFFERENCE (% D)** - The % D of the calculated amount (amount found) and the nominal amount (amount added) for each of the single component pesticides and surrogates in the PEM and Individual Mixture runs of the calibration verification on each GC column must be less than or equal to  $\pm 25.0\%$ . The % D is calculated using the following equation:

$$% D = \frac{C_{nom} - C_{calc}}{C_{nom}} \times 100$$

Where,

 $C_{\rm calc}$  = Calculated concentration of each analyte from the analyses of the standards.

 $C_{nom}$  = Nominal concentration of each analyte.

## Example

The nominal amount of gamma-BHC added to the PEM is 10 ng/mL. The calculated amount of gamma-BHC is found to be 12 ng/mL. Using the above equation, the percent difference is calculated as follows:

% D = 
$$\frac{12 - 10}{10}$$
 x 100 = 20%

## Example

The nominal amount of 4,4'-DDT added to the INDA is 10 ng/mL. The calculated amount of 4,4'-DDT is found to be 9.0 ng/mL. Using the above equation, the percent difference is calculated as follows:

% D = 
$$\frac{9.0 - 10}{10}$$
 x 100 = -10%

## Example

The nominal amount of Aldrin added to the INDB is 5.0 ng/mL. The calculated amount of Aldrin is found to be 7.0 ng/mL. Using the above equation, the percent difference is calculated as follows:

% D = 
$$\frac{7.0 - 5.0}{5.0}$$
 x 100 = 40%

#### SECTION V: BLANK CRITERIA

#### Method Required Blanks

Refer to <u>Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses</u>, Part III, Section PEST/PCB-V-B for blank data calibration criteria and the following method QC criteria:

- 1. Method Blank
- A volume of reagent water or purified solid matrix, approximate in weight or volume to the samples, which is carried through the entire analytical process to determine the levels of contamination associated with the processing and analysis of the entire set of samples. Surrogate analytes must recover between 30 150%. A method blank must be extracted and analyzed once per each SDG, or each matrix within an SDG, or each extraction procedure within an SDG, or whenever samples are extracted, whichever is most frequent.
- 2. Sulfur Cleanup
- The sulfur cleanup blank is a modified form of the method blank. It is a volume of clean solvent spiked with the surrogates and carried through the sulfur cleanup and analysis procedures. This blank is used to determine the levels of contamination associated with the separate sulfur cleanup steps. Surrogate analytes must recover between 30 150%. The sulfur cleanup blank is prepared separately when only part of a set of samples extracted together requires sulfur removal. If all of the samples associated with a given method blank require sulfur cleanup, then the method blank must be subjected to sulfur cleanup and no separate sulfur cleanup blank is required.

3. Instrument Blank -

The instrument blank is a volume of clean solvent spiked with the surrogates and analyzed on each GC column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrument analysis itself, such as the carry-over of analytes from standards or highly contaminated samples into other analyses. An instrument blank must be analyzed after a sample whose concentration exceeds the calibration range. Until an instrument blank meets the technical acceptance criteria, the system is considered contaminated.

A GPC blank, a type of instrument blank, is analyzed after the initial calibration of the GPC. It is not spiked with surrogate analytes.

#### SECTION VI: SURROGATE ANALYTE CRITERIA

Refer to <u>Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses</u>, Part III, Section PEST/PCB-VI-B for the surrogate analyte data validation criteria and the following method surrogate analyte QC criteria:

Table App.F.VI-1 - SURROGATE RETENTION TIME WINDOWS

Surrogate	Retention Time Window in Minutes
Tetrachloro-m-xylene	±0.05
Decachlorobiphenyl	±0.10

The surrogate % recovery is calculated using the following equation:

Surrogate Percent Recovery = 
$$\frac{Q_d}{Q_a} \times 100\%$$

 $Q_d$  = Quantity of surrogate determined by analysis.

Q<sub>a</sub> = Quantity of surrogate added to sample/blank.

Table App.F.VI-2 - SURROGATE SPIKE RECOVERIES

Surrogate	Percent Recovery* (Soil/Sediment and Water)
Tetrachloro-m-xylene	30-150
Decachlorobiphenyl	30-150

<sup>\*</sup> Advisory for sample analysis. Mandatory for method blanks/sulfur cleanup blanks

#### SECTION VII: PESTICIDE CLEANUP CRITERIA

Refer to <u>Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses</u>, Part III, Section PEST-VII-B for pesticide cleanup data validation criteria and the following method pesticide cleanup QC criteria:

## **GPC**

The Initial GPC Calibration consists of analyzing the GPC Calibration and GPC Calibration Verification solutions to establish the correct "Collect" and "Dump" time periods and a GPC blank to ensure that the system is free of contaminants.

1. The GPC Calibration Solution contains the following analytes in methylene chloride:

corn oil perylene bis-(2-ethylhexyl)phthalate sulfur methoxychlor

2. The GPC blank consists of methylene chloride.

## Table App.F.VII-1 - GPC CALIBRATION ACCEPTANCE CRITERIA

Peak Resolution	Corn Oil and phthalate peaks must exhibit > 85% resolution.  Phthalate and methoxychlor peaks must exhibit > 85% resolution.  Methoxychlor and perylene peaks must exhibit > 85% resolution.  Perylene and sulfur peaks must not be saturated and must exhibit >90% baseline resolution.
Peak Shape	Peaks must be observed and symmetrical for all analytes in the calibration solution.
Retention Time	The retention times must not vary more than $\pm$ 5.0% between calibrations.
Blanks	A GPC blank must be analyzed after each initial GPC calibration and target analytes cannot be present at greater than the quantitation limit for any target analyte.

The Continuing GPC Calibration consists of analyzing the following two solutions in sequence every 7 days.

1. The <u>Pesticide GPC Calibration Verification Solution</u> contains the following 6 pesticide analytes (same as matrix spiking solution) in methylene chloride:

gamma-BHC dieldrin heptachlor endrin aldrin 4,4'-DDT

2. The <u>PCB GPC Calibration Verification Solution</u> contains 2 ug/mL each of Aroclors 1016 and 1260 in methylene chloride.

## Table App.F.VII-2 - GPC CALIBRATION VERIFICATION CRITERIA

% Recovery of Single Component Pesticides	80 - 110%
Aroclor Patterns	Elution patterns must be the same as those from the Aroclor 1016 and Aroclor 1260 standards in the initial calibration sequence.

## Florisil Cartridge

The Florisil Cartridge Performance Check consists of testing every lot of Florisil prior to use or every 6 months, whichever is more frequent, by analyzing the following two solutions in a mixture.

- 1. The <u>Florisil Cartridge Check Solution</u> contains 2,4,5-trichlorophenol in acetone, at a concentration of 0.1 ug/mL.
- 2. The <u>Pesticide Spiking Solution</u> contains the following Standard Mixture A analytes at the midpoint concentration:

alpha-BHC 4,4'-DDD
heptachlor 4,4'-DDT
gamma-BHC methoxychlor
endosulfan I tetrachloro-m-xylene
dieldrin decachlorobiphenyl
endrin

#### Table App.F.VII-3 - FLORISIL CARTRIDGE PERFORMANCE CHECK CRITERIA

INDA Analyte % Recovery	80% - 120%
2,4,5-TCP Recovery	2,4,5-Trichlorophenol must recover at < 5.0%
Other Target Analytes	No interfering peaks with the target analytes

## SECTION VIII: MATRIX SPIKE/MATRIX SPIKE DUPLICATE CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-VIII-B for MS/MSD data validation criteria and the following method MS/MSD QC criteria:

A matrix spike and matrix spike duplicate must be performed for each group of samples of a similar matrix for each SDG, or each matrix within an SDG, or each group of samples of a similar concentration level.

The following advisory matrix spike analyte recoveries and RPDs are listed below:

Table App.F.VIII-1 - MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE LIMITS

	Method QC Criteria				
Analyte	Water		Soil/Sediment		
	% Recovery*	RPD**	% Recovery	RPD	
gamma-BHC (Lindane)	56-123	15	46-127	50	
Heptachlor	40-131	20	35-130	31	
Aldrin	40-120	22	34-132	43	
Dieldrin	52-126	18	31-134	38	
Endrin	56-121	21	42-139	45	
4,4'-DDT	38-127	27	23-134	50	

<sup>\*</sup>The MS/MSD % recovery is calculated using the following equation:

$$Matrix Spike Recovery = \frac{SSR - SR}{SA} \times 100$$

Where,

SSR = Spike Sample Result.

SR = Sample Result.

SA = Spike Added.

\*\*The MS/MSD relative percent difference (RPD) is calculated using the following equation:

Relative Percent Difference = 
$$\frac{|MSR - MSDR|}{1/2 (MSR + MSDR)} \times 100$$

Where,

MSR = Matrix Spike Recovery.

MSDR = Matrix Spike Duplicate Recovery.

Note: The vertical bars in the formula above indicate the absolute value of the difference, hence RPD is always positive.

## SECTION IX: FIELD DUPLICATE CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-IX-B for field duplicate data validation criteria.

## SECTION X: SENSITIVITY CHECK CRITERIA

Refer to <u>Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses</u>, Part III, Section PEST/PCB-X-B for sensitivity check data validation criteria.

#### SECTION XI: PE SAMPLES - ACCURACY CHECK CRITERIA

Refer to <u>Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses</u>, Part III, Section PEST/PCB-XI-B for accuracy check data validation criteria.

#### SECTION XII: TARGET ANALYTE IDENTIFICATION CRITERIA

Refer to <u>Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses</u>, Part III, Section PEST/PCB-XII-B for target analyte identification data validation criteria.

## SECTION XIII: ANALYTE QUANTITATION AND REPORTED QUANTITATION LIMITS CRITERIA

Refer to <u>Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses</u>, Part III, Section PEST/PCB-XIII-B for analyte quantitation and reported quantitation limit data validation criteria and the following method quantitation QC criteria:

Pesticide/PCB analytes must be quantified using the external standard method. The midpoint calibration factor from the most recent compliant initial calibration must be used to quantify sample single component pesticides on both columns and the lower sample concentration must be reported. For multicomponent analytes, the calibration factors for three to five major peaks from the most recent compliant initial calibration are each used to quantify the multicomponent analyte in the sample. The three to five concentrations are then averaged and an analyte mean concentration for the sample is calculated for each column. The lower mean concentration is reported. Pesticides/PCBs must be reported to the CRQLs listed below:

# <u>Table App.F.XIII-1 - TARGET ANALYTE LIST (TCL) AND CONTRACT REQUIRED</u> <u>QUANTITATION LIMITS (CRQLs)</u>

	Quantitation Limits			
	Water	Soil		On Column
Pesticides/Aroclors	ug/L		ug/kg	(pg)
alpha-BHC	0.050		1.7	5
beta-BHC	0.050		1.7	5
delta-BHC	0.050	1.7	5	
gamma-BHC	0.050		1.7	5
Heptachlor	0.050	1.7	5	
Aldrin	0.050		1.7	5
Heptachlor epoxid	e 0.050		1.7	5
Endosulfan I	0.050	1.7	5	
Dieldrin	0.10	3.3		10
4,4'-DDE	0.10	3.3		10
Endrin	0.10	3.3		10
Endosulfan II	0.10	3.3		10

4,4'-DDD	0.10	3.3		10	
Endosulfan sulfate	0.10	3.3		10	
4,4'-DDT	0.10	3.3		10	
Methoxychlor	0.50		17		50
Endrin ketone	0.10	3.3		10	
Endrin aldehyde	0.10	3.3		10	
alpha-Chlordane	0.050		1.7	5	
gamma-Chlordane	0.050		1.7	5	
Toxaphene	5.0		170		500
Aroclor-1016	1.0		33		100
Aroclor-1221	2.0		67		200
Aroclor-1232	1.0		33		100
Aroclor-1242	1.0		33		100
Aroclor-1248	1.0		33		100
Aroclor-1254	1.0		33		100
Aroclor-1260	1.0		33		100

**SAMPLE CONCENTRATION** - Concentrations of single component pesticides and surrogates are calculated for both GC columns using the calibration factor from the initial calibration for the midpoint concentration of the external calibration standard in the following equations:

Sample concentrations for waters:

$$ug/L = \frac{(A_x) (DF) (V_t) (GPC)}{(CF) (V_o) (V_i)}$$

Sample concentration for soils:

$$ug/Kg = \frac{(A_x) (DF) (V_t) (GPC)}{(CF) (W_s) (D) (V_i)}$$

Where,

 $A_x$  = Area of peak for the analyte being measured.

CF = Calibration Factor for the midpoint concentration from the initial calibration (area per ng).

 $V_t$  = Volume of total extract (uL).

 $V_i$  = Volume injected (uL).

 $V_o$  = Volume of sample extracted (mL).

DF = Dilution Factor. The dilution factor for analysis of water and soil/sediment samples by this method is defined as follows:

uL most conc. extract used to make dilution + uL clean solvent

uL most conc. extract used to make dilution

If no dilution is performed, DF = 1.

W<sub>s</sub> = Weight of sample extracted.

 $D = \underline{100 - \% \text{ Moisture}}$ 

100

GPC = GPC factor (If no GPC is performed, GPC = 1; if GPC is performed, then GPC = 2.0).

## Adjusted CRQL = Non-adjusted CRQL x Sample Dilution Factor

Sample concentrations for waters:

$$\begin{array}{c} \textit{Adjusted} \\ \textit{CRQL} \end{array} = \begin{array}{c} \textit{Contract} \\ \textit{CRQL} \end{array} \times \begin{array}{c} (\textit{V}_{x}) \; (\textit{V}_{t}) \; (\textit{V}_{y}) \; (\textit{DF}) \\ \hline (\textit{V}_{o}) \; (\textit{V}_{c}) \; (\textit{V}_{i}) \end{array}$$

Where,

V<sub>t</sub>, DF, V<sub>o</sub>, and V<sub>i</sub> are as given in the sample concentration equation above.

 $V_{\rm x}$  = Contract sample volume (1000 mL).

 $V_v$  = Contract injection volume (1 uL or 2 uL).

V<sub>c</sub> = Contract concentration extract volume (10,000 uL if GPC was not performed and 5,000 uL if GPC was performed).

Sample concentrations for soil/sediments:

$$\frac{\text{Adjusted}}{\text{CRQL}} = \frac{\text{Contract}}{\text{CRQL}} \times \frac{(W_x)(V_t)(V_y)(DF)}{(W_s)(V_c)(V_i)(D)}$$

Where,

V<sub>t</sub>, DF, V<sub>o</sub>, and V<sub>i</sub> are as given in the sample concentration equation above.

 $W_x$  = Contract sample weight (30 g).

 $V_v$  = Contract injection volume (1 uL or 2 uL).

V<sub>c</sub> = Contract concentrated extract volume (GPC is required: 5000 uL).

For example, the adjusted CRQL for a water sample with a 1.0U non-diluted CRQL and a 1 to 100 dilution (100 dilution factor) would be 100U, according to the following equation:

$$100U = 1.0U \times 100$$

Adjusting for dry weight, the CRQL would be calculated as:

$$Dry Wt. CRQL = \frac{\frac{Non-adjusted Weight}{100 - % Moisture}}{100}$$

For example, the dry weight CRQL for a soil sample with a 33U non-adjusted CRQL and 10% moisture would be 37U, according to the following equation:

$$37U = \frac{33U}{100 - 10}$$

The lower of the two concentrations calculated for each single component pesticide is reported on the tabulated Report Form I. For multicomponent analytes the lower  $\underline{\text{mean}}$  concentration is reported. The percent difference between the two values is reported on Form X and calculated using the following equation:

## PERCENT DIFFERENCE CALCULATION

$$& D = \frac{C_h - C_1}{C_1} \times 100$$

Where,

C<sub>h</sub> = The higher of the two concentrations for the target analyte in question.

 $C_1$  = The lower of the two concentrations for the target analyte in question.

#### SECTION XIV: SYSTEM PERFORMANCE CRITERIA

Refer to <u>Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses</u>, Part III, Section PEST/PCB-XIV-B for system performance data validation criteria.

## SECTION XV: OVERALL ASSESSMENT CRITERIA

Refer to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, Section PEST/PCB-XV-B for overall assessment data validation criteria.